Dispersion characterisation of $CaCO_3$ particles in PP/$CaCO_3$ composites

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A B S T R A C T

Polypropylene and calcium carbonate were prepared by melt extrusion in a twin screw extruder or casting in a kneader extruder. The dispersion of $CaCO_3$ particles in PP was analysed through the observation of impact-fracture surface, polished surface, cross-section surface and film using scanning electron microscopy (SEM), focused ion beam (FIB) and optical transmission microscopy (OTM). Only basic information on particle dispersion was obtained from the investigation of fracture surfaces by SEM and the FIB cross-sections by virtue of the ambiguous phase contrast and the limited area observed. However, the SEM images from polished-surfaces and the film images observed in OTM show the different particle dispersion in different samples. The derived Particle Size Distribution (PSD) curves from OTM images explain the difference of impact strength, demonstrating the relationship between particle dispersion and mechanical properties.

Keywords:
A. Polymer–matrix composites (PMCs)
D. Electron microscopy
D. Mechanical testing
Particle size distribution

1. Introduction

Polypropylene (PP) is one of the most widely used engineering plastics with good mechanical properties, processability, excellent recyclability, good heat stability and chemical resistance. However, low modulus, notch sensitive features, and brittleness under low temperatures or high strain rates limit its applications [1–4]. The addition of rigid inorganic fillers into PP is a common practice in the plastics industry reducing production costs and improving stiffness, toughness, hardness, dimensional stability, and heat performance [1,5–7].

$CaCO_3$ particles have been widely compounded into polymer matrices to enhance the impact toughness of polymers [1,8–10]. By examining the fracture surface using SEM [11–20], ultrathin sections using TEM [21–27] or grinded surfaces using BSE-detectors [28], researchers usually agree that the improved impact strength depends on the particle dispersion such as particle shape, particle size and aspect ratio, owing to their great impact on the particle–matrix interaction and the overall structure. Good dispersion of $CaCO_3$ in a polymer matrix occurs when particles are evenly dispersed without agglomeration. However, $CaCO_3$ particles tend to agglomerate because of their high hydrophilicity and high surface energy. The finer the particles of $CaCO_3$ are, the more likely they are to agglomerate.

In composites containing particles of different size and shape, the PSD and aspect ratio are often used to describe particle dispersion. For most PP/$CaCO_3$ composites, $CaCO_3$ particles have a block-like shape, which can be considered near-spherical, and particle size is the best one to accurately describe the dispersion of $CaCO_3$ particles in polymer. The particle distribution may be estimated by the visualisation in SEM/TEM images. Although the phase contrast in SEM measurements may be sufficient to distinguish the polymer and particles, it remains difficult to measure accurately the size of particles. TEM images may improve the phase comparison but agglomerates are easily missed due to the limited area examined and sample preparation is time-consuming.

Here, we report a study of the dispersion of different-sized $CaCO_3$ particles in PP, in which the effect of particle size on mechanical properties, crystallisation and morphologies was investigated using X-ray Diffraction (XRD), SEM, FIB and OTM. A 'macro-method' was developed to describe the $CaCO_3$ particle dispersion in PP using PSD, to give an insight into the relationship between particle dispersion and mechanical properties.

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